#### Reference

Belshay, T.I. 2017. Magnitude of Residues of Sulfoxaflor in Nectar, Pollen, and Whole Plants Following Foliar Application of GF-2372 to Alfalfa. Unpublished study performed by Smithers Viscient, Snow Camp, North Carolina. Laboratory Project ID: 14050.4117. Study sponsored by Dow AgroSciences LLC, Indianapolis, Indiana. Study completed December 5, 2017.

#### 1. STUDY INFORMATION

**Chemical:** Sulfoxaflor **PC Code:** 005210

**Test Material:** Transform®WG **Purity:** 49.4% (w:w) ai (494 g ai/kg) **Study Type:** Non-guideline field residue study on alfalfa to establish sulfoxaflor and various

metabolite levels in whole flowers, pollen, and nectar following two foliar

(Trial 01): Tidewater Agronomics, Inc., 313

Turnpike Road, Belvidere, North Carolina 27919

**(Trial 02):** Turner Ag Research, 1233 E. Beamer Street, Suite E, Woodland, California 95776

(Analytical): Smithers Viscient, MRC, 790 Main

Street, Wareham, Massachusetts 02571

applications.

Sponsor: Dow AgroSciences LLC Performing Laboratories:

9330 Zionsville Road

Indianapolis, Indiana 46268

**Report Number:** 14050.4117

Completion

Date: December 5, 2017

**Experiment** 

Study

**Start/End Date:** July 15, 2016 to September

30, 2016 (field phase)

**Study Location:** 2 Field Trials:

Hertford, North Carolina (14050.4117-01); Live Oak, California (14050.4117-02)

**GLP Status:** GLP-compliant; 40 CFR Part 160

#### 2. REVIEWER INFORMATION

Primary Reviewer: Christie E. Padova, B.S., Environmental Scientist, CDM/CSS-Dynamac JV

Signature: Christic E. Padora

**Date:** 03/21/17

Secondary Reviewer: Keith Sappington, Senior Science Advisor, OPP/EFED/ERB5

Signature:

**Date:** 7-10-19

This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac JV personnel. The CDM/CSS-Dynamac Joint Venture role does not include establishing Agency policies.

#### 3. EXECUTIVE SUMMARY

This study was designed to measure the magnitude of residues of sulfoxaflor and its four major metabolites, X11579457, X11719474, X11519540 and X11721061, in alfalfa (*Medicago sativa*) whole plant, nectar and pollen, which represent potential exposure risks to pollinators in the field. Two separate trials were conducted, at locations in North Carolina (Trial 1) and California (Trial 2). Three subplots at each trial location received two foliar applications of Transform® WG at 0.090 lb ai/A/application, based on a maximum seasonal rate of 0.186 lb ai/A, applied in two application timings at the minimum retreatment interval of 7 days. Whole plants were collected from each site prior to treatment, and whole plant and flower samples (for nectar and pollen) were collected from early- through late-bloom for residue analysis (0 through 14 Days After Last Application [DALA]). Samples were collected and analyzed by validated analytical methods to determine the residue concentrations.

#### A summary of the key findings is as follows:

- 1. Two foliar applications to alfalfa plants at 0.090 lb ai/A/application (based on a maximum seasonal rate of 0.186 lb ai/A), yielded detectable residues of sulfoxaflor in nectar, pollen and whole plant at both trial sites.
- 2. In alfalfa plant matrices, total sulfoxaflor residues (TSR) were greatest for each trial site in pollen, followed by nectar and then whole plant matrices, and measured residues were greater in the California trial (Trial 2) compared to North Carolina (Trial 1). In California, maximum mean TSR levels were 87.0, 21.8, and 8.92 mg/kg in pollen, nectar, and whole plant matrices, respectively, and in North Carolina, maximum mean TSR levels were 41.9, 11.5, and 6.05 mg/kg, respectively.
- 3. In nectar and whole plant samples, parent sulfoxaflor accounted for the majority of TSR while in pollen samples, sulfoxaflor accounted for the majority of TSR at the California trial, and metabolite X11719474 accounted for the majority of TSR at the North Carolina trial. Maximum mean concentrations of sulfoxaflor observed at the California trial (0 DALA) were 58.4 mg/kg in pollen, 19.8 mg/kg in nectar, and 6.89 mg/kg in whole plant. Maximum mean concentrations of sulfoxaflor at the North Carolina trial (0 DALA) were 7.67, 10.3, and 4.83 mg/kg in pollen, nectar, and whole plant samples, respectively. The major metabolite, X11719474, was present at maximum mean concentrations of 33.7 and 27.6 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively, and ranged from maximum means of 1.14 to 2.30 mg/kg in nectar and whole plant samples from both trials (0 to 2 DALA). Metabolite X11519540 was present at maximum mean concentrations of 0.394 and 0.641 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively. Metabolites X11579457 and X11721061 were found at maximum mean concentrations of 0.195 mg/kg and 0.142 mg/kg, respectively, in pollen from the California trial (each at 0 DALA).

Analyte	Matrix	Maximum Measured Sulfoxaflor Concentration (mg/kg)	Study Site	Maximum Average Sulfoxaflor Concentration (mg/kg)	Study Site
Sulfoxaflor	Pollen	73.6	California	58.4	California
	Nectar	31.8	California	19.8	California
	Whole Plant	8.66	California	6.89	California
X11579457	Pollen	0.233	California	0.195	California

Analyte	Matrix	Maximum Measured Sulfoxaflor Concentration (mg/kg)	Study Site	Maximum Average Sulfoxaflor Concentration (mg/kg)	Study Site
	Nectar	0.0299	California	0.0159	California
	Whole Plant	0.0161	California	0.0137	California
X11719474	Pollen	35.3	California	33.7	North Carolina
	Nectar	5.22	California	1.80	California
	Whole Plant	2.53	California	2.30	California
X11519540	Pollen	0.751	California	0.641	California
	Nectar	0.142	California	0.109	California
	Whole Plant	0.0487	California	0.0460	California
X11721061	Pollen	0.195	California	0.142	California
	Nectar	0.0630	California	0.0512	California
	Whole Plant	0.102	California	0.0876	California

- 4. Trends in sulfoxaflor and total sulfoxaflor residue (TSR) concentrations declined in alfalfa pollen, nectar, and whole plant samples from the early- and early-mid bloom period (0-2 DALA) to the late-bloom period (14 DALA) at both trial locations.
- 5. The  $DT_{50}$  values for sulfoxaflor in nectar were 0.37 (NC site) and 1.2 days (CA site). The  $DT_{50}$  values for sulfoxaflor in pollen were 0.26 (NC site) and 2.3 days (CA site).

#### 4. STUDY VALIDITY

4. STUDY VALIDITY	
Guideline Followed:	Non-guideline study
<b>Guideline Deviations:</b>	N/A
Other Deviations:	Problems with out of range QC sample recovery. With two sample recoveries of 194 and 546% of nominal at the LOQ spike concentration. While this is a deviation, over estimation of sample residue would be protective of a lower value and these deviations affect values near the LOQ.  Only two sites were evaluated, whereas USEPA (2016) recommends a minimum of 3 sites/regions within the growing area. Therefore,
	variability in residue values associated with geographic differences among growing regions may be underestimated.
Classification:	Supplemental
Rationale:	No major deviations were identified in this study that would affect the scientific integrity of this study.
Reparability:	N/A

#### 5. MATERIALS AND METHODS

Test Material Characterization						
Test item: Synonyms:	Transform® WG (GF-2372) Isoclast™ (ai), XDE-208 (ai)	CAS #:	946578-00-3			
Description:	Water-dispersible granule	Purity:	49.4% (w:w) sulfoxaflor (ai)			
Lot No./Batch No.	2E31160A63	Density:	Not Reported			

Material Source:	Not reported	Cert.#	Not reported
Material Receipt		Analysis	
Date:	Not reported	Date:	Not reported
Expiration Date:	November 8, 2018	Solubility:	Not Reported
Storage of Test Mat'l:	Not reported	Sample	
		Storage:	Not reported

#### 5A. STUDY DESIGN

This study was conducted to quantify the magnitude and decline of residues of sulfoxaflor and its major metabolites – X11579457, X11719474, X11519540 and X11721061 – in alfalfa (*Medicago sativa*) matrices following two foliar application of Transform® WG at 0.090 lb ai/A/application to field plots planted to alfalfa in North Carolina (Trial 1) and California (Trial 2). The first application was made at BBCH 60-61, with a retreatment interval of 7 days and 10 days for Trials 1 and Trial 2, respectively. Test plots were divided into three replicate areas (A, B, and C), each measuring 120 feet x 19-20 feet. A control plot was not included in the study design; control of experimental bias was achieved through replication within the test item treatment groups. Whole plant samples were collected prior to the first treatment (-7 DALA for the North Carolina trial and -10 DALA for the California trial), and whole plants, nectar, and pollen samples were collected 0, 1, 2, 7 and 14 DALA. The four metabolites of interest were X11579457 (5-[1-(S-methylsulfonimidoyl)ethyl]-2-(trifluoromethyl)pyridine), X11719474 (N-(methyl(oxido){1-[6-(trifluoromethyl)pyridine), and X11721061 (1-[6-(trifluoromethyl)pyridine-3-yl]ethanol). Soil samples were not collected. These data can be used to quantify the potential dietary exposure to pollinators in the field.

#### **5B. APPLICATION TIMING AND RATES**

Transform® WG, a wettable granular formulation containing 49.4% sulfoxaflor, was applied twice to foliage of commercial varieties/cultivars of alfalfa at 0.090 lb ai/A/application (101 g ai/ha/application). The first application occurred prior to bloom (at BBCH 60-61), with the retreatment application (7- or 10-day retreatment interval) during early bloom (at BBCH 62-63) via boom sprayers at spray volumes of 16.0 to 20.2 gal/A (99.2-100.9% of target). Information on the application rates and timing of applications is provided in **Table 1.** 

Table 1. Summary of alfalfa study site characteristics (treated sites only).

Attribute	Site 1 (14050-4117-01)	Site 2 (14050-4117-02)
	Hertford, North Carolina	Live Oak, California
Variety	N/A	N/A
Transplant Date	N/A	N/A
Application Dates	App 1: May 3, 2017	App 1: August 18, 2016
	App 2: May 10, 2017	App 2: August 28, 2016
Air Temp (°F)	App 1: 70	App 1: 63
	App 2: 65	App 2: 68
Humidity (%)	App 1: 55	App 1: 88
	App 2: 92	App 2: 42
Wind speed	App 1: 3-6/ S	App 1: 0.4-0.6/N
(mph)/direction	App 2: 0-2/SE	App 2: 0.4-1.1 /N
Timing	App 1: prior to bloom	App 1: prior to bloom

Attribute	Site 1 (14050-4117-01)	Site 2 (14050-4117-02)
	Hertford, North Carolina	Live Oak, California
	App 2: during bloom	App 2: during bloom
BBCH Growth Stage	App 1: 60-61	App 1: 60
	App 2: 62-63	App 2: 63
Spray Volume (gal/A)	App 1: 16.0	App 1: 20.1
	App 2: 16.3	App 2: 20.2
Rate (lb ai/A)	App 1: 0.0893	App 1: 0.0900
	App 2: 0.0902	App 2: 0.0908
Soil Type	Loamy sand	Loam
OM (%)	1.7-1.8	2.2
рН	6.4-6.5	6.8
CEC (meq/100g)	5.3-5.4	13.5
Sand/Silt/Clay (%)	84-86/12-14/2	47/28/25

#### **5C. STUDY SITE LOCATION AND CHARACTERISTICS**

A summary of application, soil, and meteorological data from the two study sites is shown in **Table 1.** Trials were conducted on planted plots of loamy sand (North Carolina) or loam (California) soils. Soil organic matter varied from 1.7 to 2.2% for each trial. Standard agronomic practices for growing alfalfa were used on the treated test plots; no maintenance pesticides were used at either trial. Overall, the weather conditions did not negatively impact the crop growth or development. For the North Carolina trial, the average minimum and maximum air temperatures were 58.53 to 76.51°F and 71.00 to 91.00 °F during the months of April and May 2017, respectively. No supplemental irrigation was needed during the North Carolina trial, which received 0.11 and 6.05 inches of rainfall during the months of April and May 2017, respectively. For the California trial, the average minimum and maximum air temperatures were 60.7 to 92.7°F and 55.0 to 91.1°F during the months of August and September 2016, respectively. Nine inches of flood-irrigation well water was provided during the month of August at the California trial, which received no rainfall during the months of August and September, 2016.

#### **5D. Sample Collection, Handling, Processing**

#### **Plant Matrices:**

For each matrix, one sample was collected from each replicate plot A, B and C. Whole plant samples were collected 0, 1, 2, 7, and 14 DALA, encompassing early (x2), early-mid, mid, and late bloom periods. A single untreated control sample of whole plants was also collected prior to the first application (-7 DALA for the North Carolina trail and -10 DALA for the California trial). Alfalfa plants were indiscriminately-selected, hand-clipped and trimmed of fruit and flowers, and double-bagged in sealable gallon-size plastic bags. The target weight for whole plants was 500 g. Samples were placed in a cooler on substitute ice, transported to the field station, and placed in frozen storage.

Whole flowers – for the purpose of extracting nectar and pollen – were collected at 0, 1, 2, 7, and 14 DALA. Flowers were indiscriminately-selected throughout the plots and all areas of the alfalfa plants; care was taken not to cross-contaminate between replicate plots. Samples were sealed in plastic bags and transported in coolers on substitute ice to the field laboratory for processing. At the field station, nectar was extracted from whole flowers using centrifugation and collected directly into capillary tubes.

Additional whole flowers were dried overnight, and the pollen was dislodged by hand-tapping onto black tiles and then transferred to sample vials. The target weight for nectar and pollen samples was 100 mg.

Samples (whole plants, nectar and pollen) were maintained frozen until shipped overnight via commercial freezer trucks to the analytical laboratory in Wareham, Massachusetts.

**Soil.** Soil samples were not collected for residue analysis.

Sample storage and transport: All sample matrices (whole plant, nectar, and pollen) were shipped frozen overnight from the field stations to the analytical laboratory, where they were received frozen or cold and in good condition. All samples were subsequently stored frozen (-25 to -10°C) at the analytical laboratory prior to extraction and analyses. Whole plant samples were macerated with dry ice to obtain a homogeneous sample prior to extraction. Whole plant, nectar, and pollen samples from the North Carolina trial were stored frozen for up to 2.5, 2.1, and 2.1 months prior to analysis, respectively. Whole plant, nectar, and pollen samples from the California trial were stored frozen for up to 10.6, 10.0, and 10.0 months prior to analyses, respectively. Tank mixes were also sampled at each application date and stored frozen for up to 5.8 months for the North Carolina trial or 14.2 months for the California trial prior to analysis.

#### **5E. ANALYTICAL METHODS**

The residues of sulfoxaflor and its major metabolites X11579457, X11719474, X11519540, and X11721061 were determined in whole plant, pollen and nectar samples using liquid chromatography/mass spectrometry (LC/MS/MS). Details of the analytical methods are provided in the study report. Residues were extracted from pollen, nectar, and whole plant samples with acetonitrile:purified water (80:20, v:v), and extracts were concentrated under nitrogen followed by enzymatic deconjugation, and cleaned-up with solid phase extraction (SPE). The method was validated by fortification of pollen, nectar, whole plant, and 50% sucrose solution matrices with sulfoxaflor, X11579457, X11719474, X11721061, and X11519540 at the LOD (*ca.* 1/3 of the LOQ), the LOQ, and high-level concentrations of 100X, 2000X, or 1000X the LOQ (Smithers Viscient Study Nos. 14050.6275 and 14050.6268). For metabolites X11579457 and X11519540, pollen matrix effects were observed to be >20%; therefore, matrix-matched standards were used for these analyses.

#### **5F. QUALITY ASSURANCE**

Freezer Stability. The frozen storage stability of sulfoxaflor and its metabolites X11719474 and X11721061 were demonstrated in various crop matrices (orange whole fruit, peach whole fruit, wheat grain, and soybean seed) for at least 680 days (22.4 months) when stored at *ca.* -20°C (MRID 47832224). Frozen storage stability was also demonstrated in sunflower pollen and nectar stored frozen at ≤-18°C for at least 9 months (Dow AgroSciences Study No. 150537). It was reported an additional storage stability study is on-going to determine stability of the metabolites in pollen and nectar.

**Transit Stability:** Transit stability samples were not included in the study design.

**Spike Recoveries.** The performance of the analytical method for determination of sulfoxaflor and metabolite residues in alfalfa matrices was determined with each set of field samples by fortifying

aliquots of appropriate control matrix (obtained prior to treatment) with a mixed solution of sulfoxaflor and its metabolites (QC samples). Control pollen and whole plants samples were fortified with sulfoxaflor and metabolites (X11579457, X11719474, X11519540, and X11721061) at 0 mg/kg (control), 0.0030 mg/kg (LOD), 0.0100 mg/kg (LOQ), 10.0 mg/kg (1000X LOQ), or 50.0 mg/kg (5000X LOQ). Control nectar samples were fortified with sulfoxaflor at 0 (control), 0.000300 mg/kg (LOD), 0.00100 mg/kg (LOQ), 1.00 mg/kg (1000X LOQ), or 50.0 mg/kg (50,000X LOQ), or with metabolites (X11579457, X11719474, X11519540, and X11721061) at 0 mg/kg (control), 0.00300 mg/kg (LOD), 0.0100 mg/kg (LOQ), 1.00 mg/kg (100X LOQ), or 50.0 mg/kg (5000X LOQ). Samples fortified at the LOD demonstrated that observable peaks at the LOD could be distinguished from untreated control samples; results were not included for average percent recoveries. The limits of detection and quantification for each analyte during the analytical phase are provided in Table 2.

Table 2. Method LOQ and LOD in each matrix.

Analyte	Matrix	LOQ (mg/kg)	LOD (mg/kg)
Sulfoxaflor	Whole plant	0.0100	0.00300
	Pollen	0.0100	0.00300
	Nectar	0.00100	0.000300
X11579457, X11719474,	Whole plant	0.0100	0.00300
X11519540, X11721061	Pollen	0.0100	0.00300
	Nectar	0.0100	0.00300

#### 6. RESULTS:

#### **6A. QUALITY ASSURANCE RESULTS**

**Transit Stability.** Transit stability samples were not included in the study design.

Spike Recoveries. The individual QC recoveries for all analytes generally fell within the range of 70 to 120%; recoveries outside this range were evaluated on a case-by-case basis, and were included in statistical calculations if deemed not to be outliers. Relative standard deviations at each level were all less than 20%, unless noted below. Results from laboratory spiked QC samples are summarized in Table 3.

**Table 3. Concurrent Recoveries.** 

Analyte	Matrix	Fortification Levels (ng/g)	Recovery Range (%)	Mean Recovery (% ± SD)	RSD (%)	n
Sulfoxaflor	Whole plant	0.0100/1.00/20.0	61.4 – 141	94.7 ± 20.7	21.8	16
	Pollen	0.0100/9.80/50.0 66.0 – 123 86.6 ± 16		86.6 ± 16.1	18.6	16
	Nectar	0.00100/1.00/50.0	73.4 – 164	104.3 ± 24.2	23.2	14
X11579457	Whole plant	0.0100/1.00/20.0	71.4 – 123	92.6 ± 14.7	15.9	16
	Pollen	0.0100/9.80/50.0	72.4 – 133	105.5 ± 15.7	14.9	16
	Nectar	0.0100/1.00/50.0	62.1 – 113	92.3 ± 15.8	17.2	13
X11719474	Whole plant	0.0100/1.00/20.0	103 – 132	116.1 ± 8.2	7.1	16
	Pollen	0.0100/9.80/50.0	73.6 – 135	103.7 ± 18.0	17.3	16
	Nectar	0.0100/1.00/50.0	98.5 – 129	112.0 ± 8.5	7.6	15

Analyte	Matrix	Fortification Levels (ng/g)	Recovery Range (%)	Mean Recovery (% ± SD)	RSD (%)	n
X11519540	Whole plant	0.0100/1.00/20.0	70.1 – 110	92.6 ± 12.6	13.6	16
	Pollen	0.0100/9.80/50.0	82.2 – 140	103.2 ± 16.1	15.6	16
	Nectar	0.0100/1.00/50.0	49.9 – 115	85.7 ± 23.8	27.7	15
X11721061	Whole plant	0.0100/0.925/20.0	77.7 – 123	95.5 ± 12.4	13.0	16
	Pollen	0.0100/9.80/50.0	76.5 – 122	93.4 ± 12.5	13.4	16
	Nectar	0.0100/1.00/50.0	85.1 – 122	104.6 ± 8.8	8.4	15

#### 6B. Magnitude of Residues in Bee-Relevant Matrices

**Alfalfa Nectar, Pollen, and Whole Plant.** Summary statistics of the overall magnitude of sulfoxaflor, X11579457, X11719474, X11519540, X11721061, and total sulfoxaflor residues (TSR) are shown in Tables 4-7. In nectar and whole plant samples, parent sulfoxaflor accounted for the majority of TSR while in pollen samples, sulfoxaflor accounted for the majority of TSR at the California trial, and metabolite X11719474 accounted for the majority of TSR at the North Carolina trial. Concentrations of residues were higher in California relative to North Carolina, and were found at greatest concentrations in pollen, followed by nectar, and then whole plant tissue. Maximum mean concentrations of sulfoxaflor observed at the California trial (0 DALA) were 58.4 mg/kg in pollen, 19.8 mg/kg in nectar, and 6.89 mg/kg in whole plant. Maximum mean concentrations of sulfoxaflor at the North Carolina trial (0 DALA) were 7.67, 10.3, and 4.83 mg/kg in pollen, nectar, and whole plant samples, respectively. The major metabolite, X11719474, was present at maximum mean concentrations of 33.7 and 27.6 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively, and ranged from maximum means of 1.14 to 2.30 mg/kg in nectar and whole plant samples from both trials (0 to 2 DALA). Metabolite X11519540 was present at maximum mean concentrations of 0.394 and 0.641 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively. Metabolites X11579457 and X11721061 were found at maximum mean concentrations of 0.195 mg/kg and 0.142 mg/kg, respectively, in pollen from the California trial (each at 0 DALA).

Table 4. Maximum analyte residues recovered from alfalfa pollen, nectar, and whole plant across all sampling dates.

Trial Site	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)	
		Poll	en from Flowe	rs			
Hertford, NC	8.91	0.120	34.7	0.403	0.0558	43.8	
Live Oak, CA	73.6	0.233	35.3	0.751	0.195	110	
		Nec	tar from Flowe	rs			
Hertford, NC	12.8	<loq< td=""><td>1.48</td><td>0.0368</td><td>0.0168</td><td>13.9</td></loq<>	1.48	0.0368	0.0168	13.9	
Live Oak, CA	31.8	0.0299	5.22	0.142	0.0630	32.1	
	Whole Plant						
Hertford, NC	5.61	<loq< td=""><td>1.26</td><td>0.0396</td><td>0.0165</td><td>6.83</td></loq<>	1.26	0.0396	0.0165	6.83	
Live Oak, CA	8.66	0.0161	2.53	0.0487	0.102	9.92	

 $\label{eq:log_log_log} LOQ/LOD = 0.00100/0.000300 \; mg/kg \; for \; sulfox aflor \; in \; nectar; \; LOQ/LOD = 0.0100/0.00300 \; mg/kg \; for \; all \; remaining \; analytes in \; all \; remaining \; matrices.$ 

Table 5. Maximum mean analyte residues recovered from alfalfa pollen, nectar, and whole plant across

all sampling dates.

Trial Site	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)	
		Poll	en from Flowe	rs			
Hertford, NC	7.67	0.105	33.7	0.394	0.0451	41.9	
Live Oak, CA	58.4	0.195	27.6	0.641	0.142	87.0	
		Nec	tar from Flowe	rs			
Hertford, NC	10.3	<loq< td=""><td>1.14</td><td>0.0337</td><td>0.0145</td><td>11.5</td></loq<>	1.14	0.0337	0.0145	11.5	
Live Oak, CA	19.8	0.0159	1.80	0.109	0.0512	21.8	
	Whole Plant						
Hertford, NC	4.83	<loq< td=""><td>1.17</td><td>0.0341</td><td>0.0130</td><td>6.05</td></loq<>	1.17	0.0341	0.0130	6.05	
Live Oak, CA	6.89	0.0137	2.30	0.0460	0.0876	8.92	

LOQ/LOD = 0.00100/0.000300 mg/kg for sulfoxaflor in nectar; LOQ/LOD = 0.0100/0.00300 mg/kg for all remaining analytes in all remaining matrices.

Table 6. Mean (min, max) concentrations of analytes in alfalfa pollen, nectar, and whole plants in Hertford, North Carolina (Trial 14050-4117-01).

DALA	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)	
	Pollen from Flowers						
0	7.67 (5.55, 8.91)	0.105 (0.0932, 0.120)	33.7 (32.8, 34.7)	0.394 (0.388, 0.403)	0.0451 (0.0389, 0.0558)	41.9 (39.6, 43.8)	
1	0.525 (0.484, 0.598)	<loq (<loq,="" <loq)<="" td=""><td>2.36 (1.84, 2.98)</td><td>0.0390 (0.0336, 0.0493)</td><td>0.0169 (0.0131, 0.0211)</td><td>2.94 (2.49, 3.54)</td></loq>	2.36 (1.84, 2.98)	0.0390 (0.0336, 0.0493)	0.0169 (0.0131, 0.0211)	2.94 (2.49, 3.54)	
2	0.148 (0.126, 0.182)	<loq (<lod,="" <loq)<="" td=""><td>0.649 (0.602, 0.692)</td><td>0.0202 (0.0194, 0.0207)</td><td><loq (<loq,="" <loq)<="" td=""><td>0.826 (0.807, 0.860)</td></loq></td></loq>	0.649 (0.602, 0.692)	0.0202 (0.0194, 0.0207)	<loq (<loq,="" <loq)<="" td=""><td>0.826 (0.807, 0.860)</td></loq>	0.826 (0.807, 0.860)	
7	0.0193 (0.017, 0.0219)	<lod (<lod,="" <lod)<="" td=""><td>0.101 (0.0999, 0.102)</td><td><lod (<lod,="" <lod)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.127 (0.124, 0.131)</td></loq></td></lod></td></lod>	0.101 (0.0999, 0.102)	<lod (<lod,="" <lod)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.127 (0.124, 0.131)</td></loq></td></lod>	<loq (<lod,="" <loq)<="" td=""><td>0.127 (0.124, 0.131)</td></loq>	0.127 (0.124, 0.131)	
14	<loq (<lod,="" <loq)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0435 (0.0352, 0.0562)</td><td><lod (<lod,="" <lod)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.0530 (0.0412, 0.0657)</td></loq></td></lod></td></lod></td></loq>	<lod (<lod,="" <lod)<="" td=""><td>0.0435 (0.0352, 0.0562)</td><td><lod (<lod,="" <lod)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.0530 (0.0412, 0.0657)</td></loq></td></lod></td></lod>	0.0435 (0.0352, 0.0562)	<lod (<lod,="" <lod)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.0530 (0.0412, 0.0657)</td></loq></td></lod>	<loq (<lod,="" <loq)<="" td=""><td>0.0530 (0.0412, 0.0657)</td></loq>	0.0530 (0.0412, 0.0657)	
			Nectar from F	lowers			
0	10.3 (8.97, 12.8)	<loq (<loq,="" <loq)<="" td=""><td>1.14 (0.952, 1.48)</td><td>0.0337 (0.0299, 0.0368)</td><td>0.0145 (0.0121, 0.0168)</td><td>11.5 (10.1, 13.9)</td></loq>	1.14 (0.952, 1.48)	0.0337 (0.0299, 0.0368)	0.0145 (0.0121, 0.0168)	11.5 (10.1, 13.9)	
1	1.45 (1.04, 2.12)	<loq (<lod,="" <loq)<="" td=""><td>0.228 (0.114, 0.377)</td><td>0.00963 (<loq, 0.0136)</loq, </td><td><loq (<lod,="" <loq)<="" td=""><td>1.69 (1.16, 2.52)</td></loq></td></loq>	0.228 (0.114, 0.377)	0.00963 ( <loq, 0.0136)</loq, 	<loq (<lod,="" <loq)<="" td=""><td>1.69 (1.16, 2.52)</td></loq>	1.69 (1.16, 2.52)	
2	0.526 (0.493, 0.581)	<lod (<lod,="" <lod)<="" td=""><td>0.144 (0.0853, 0.179)</td><td><loq (<loq,="" <loq)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.678 (0.598, 0.768)</td></lod></td></loq></td></lod>	0.144 (0.0853, 0.179)	<loq (<loq,="" <loq)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.678 (0.598, 0.768)</td></lod></td></loq>	<lod (<lod,="" <lod)<="" td=""><td>0.678 (0.598, 0.768)</td></lod>	0.678 (0.598, 0.768)	
7	0.0152 (0.0118, 0.0215)	<lod (<lod,="" <lod)<="" td=""><td>0.0499 (0.0469, 0.0557)</td><td><lod (<lod,="" <lod)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0697 (0.0638, 0.0732)</td></lod></td></lod></td></lod>	0.0499 (0.0469, 0.0557)	<lod (<lod,="" <lod)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0697 (0.0638, 0.0732)</td></lod></td></lod>	<lod (<lod,="" <lod)<="" td=""><td>0.0697 (0.0638, 0.0732)</td></lod>	0.0697 (0.0638, 0.0732)	
14	<l0q (<l0q,="" <l0q)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0571 (0.0443, 0.0813)</td><td><lod (<lod,="" <lod)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0621 (0.0493, 0.0863)</td></lod></td></lod></td></lod></td></l0q>	<lod (<lod,="" <lod)<="" td=""><td>0.0571 (0.0443, 0.0813)</td><td><lod (<lod,="" <lod)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0621 (0.0493, 0.0863)</td></lod></td></lod></td></lod>	0.0571 (0.0443, 0.0813)	<lod (<lod,="" <lod)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td>0.0621 (0.0493, 0.0863)</td></lod></td></lod>	<lod (<lod,="" <lod)<="" td=""><td>0.0621 (0.0493, 0.0863)</td></lod>	0.0621 (0.0493, 0.0863)	

DALA	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)
			Whole Pla	nt		
0	4.83 (3.84, 5.61)	<loq (<loq,="" <loq)<="" td=""><td>1.17 (1.09, 1.26)</td><td>0.0341 (0.0297, 0.0396)</td><td>0.0130 (0.0111, 0.0165)</td><td>6.05 (4.98, 6.83)</td></loq>	1.17 (1.09, 1.26)	0.0341 (0.0297, 0.0396)	0.0130 (0.0111, 0.0165)	6.05 (4.98, 6.83)
1	0.525 (0.417, 0.622)	<lod (<lod,="" <lod)<="" td=""><td>0.166 (0.0551, 0.234)</td><td>0.00970 (<loq, 0.0123)</loq, </td><td><loq (<loq,="" <loq)<="" td=""><td>0.707 (0.484, 0.850)</td></loq></td></lod>	0.166 (0.0551, 0.234)	0.00970 ( <loq, 0.0123)</loq, 	<loq (<loq,="" <loq)<="" td=""><td>0.707 (0.484, 0.850)</td></loq>	0.707 (0.484, 0.850)
2	0.328 (0.276, 0.376)	<lod (<lod,="" <lod)<="" td=""><td>0.147 (0.110, 0.173)</td><td><loq (<loq,="" 0.0101)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.489 (0.398, 0.552)</td></loq></td></loq></td></lod>	0.147 (0.110, 0.173)	<loq (<loq,="" 0.0101)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.489 (0.398, 0.552)</td></loq></td></loq>	<loq (<loq,="" <loq)<="" td=""><td>0.489 (0.398, 0.552)</td></loq>	0.489 (0.398, 0.552)
7	0.134 (0.092, 0.219)	<loq (<lod,="" <loq)<="" td=""><td>0.409 (0.261, 0.512)</td><td><loq (<loq,="" <loq)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.557 (0.364, 0.746)</td></loq></td></loq></td></loq>	0.409 (0.261, 0.512)	<loq (<loq,="" <loq)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.557 (0.364, 0.746)</td></loq></td></loq>	<loq (<loq,="" <loq)<="" td=""><td>0.557 (0.364, 0.746)</td></loq>	0.557 (0.364, 0.746)
14	0.00997 ( <loq, 0.0143</loq, 	<lod (<lod,="" <loq)<="" td=""><td>0.439 (0.313, 0.549)</td><td><lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.458 (0.326, 0.575)</td></loq></td></lod></td></lod>	0.439 (0.313, 0.549)	<lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.458 (0.326, 0.575)</td></loq></td></lod>	<loq (<loq,="" <loq)<="" td=""><td>0.458 (0.326, 0.575)</td></loq>	0.458 (0.326, 0.575)

LOQ/LOD = 0.00100/0.000300 mg/kg for sulfoxaflor in nectar; LOQ/LOD = 0.0100/0.00300 mg/kg for all remaining analytes in all remaining matrices.

Table 7. Mean (min, max) concentrations of analytes in alfalfa pollen, nectar, and whole plants in Live Oak, California (Trial 14050-4117-02).

DALA	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)
			Pollen from Fl	owers		
0	58.4 (41.9, 73.6)	0.195 (0.122, 0.233)	27.6 (13.5, 35.3)	0.641 (0.577, 0.751)	0.142 (0.113, 0.195)	87.0 (74.2, 110)
1	49.9 (39.2, 68.9)	0.128 (0.0905, 0.159)	20.6 (16.8, 24.4)	0.480 (0.341, 0.739)	0.103 (0.0925, 0.121)	71.2 (56.6, 94.3)
2	26.8 (17.4, 35.0)	0.0912 (0.0854,	11.2 (9.55, 13.5)	0.246 (0.193, 0.289)	0.0673 (0.0624,	38.4 (28.1, 49.0)
		0.101)			0.0715)	
7	10.5 (6.89, 14.9)	0.0354 (0.0271,	5.33 (3.48, 8.63)	0.0898 (0.0404,	0.0219 (0.0202,	16.0 (10.5, 23.7)
		0.0469)		0.143)	0.0238)	
14	0.259 (0.0880, 0.504)	<loq (<loq,="" <loq)<="" td=""><td>0.561 (0.227, 0.852)</td><td><loq (<loq,="" <loq)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.833 (0.703, 1.05)</td></loq></td></loq></td></loq>	0.561 (0.227, 0.852)	<loq (<loq,="" <loq)<="" td=""><td><loq (<lod,="" <loq)<="" td=""><td>0.833 (0.703, 1.05)</td></loq></td></loq>	<loq (<lod,="" <loq)<="" td=""><td>0.833 (0.703, 1.05)</td></loq>	0.833 (0.703, 1.05)

DALA	Sulfoxaflor (mg/kg)	X11579457 (mg/kg)	X11719474 (mg/kg)	X11519540 (mg/kg)	X11721061 (mg/kg)	Total Sulfoxaflor Residues (TSR) (mg/kg)		
	Nectar from Flowers							
0	19.8 (10.6, 31.8)	0.0159 ( <lod, 0.0299)</lod, 	1.80 (0.0735, 5.22)	0.109 (0.0775, 0.142)	0.0443 (0.0292, 0.0584)	21.8 (16.0, 32.1)		
1	14.3 (8.53, 17.5)	<lod (<lod,="" <loq)<="" td=""><td>0.182 (0.0823, 0.343)</td><td>0.0426 (0.0257, 0.0613)</td><td>0.0512 (0.0313, 0.0630)</td><td>14.6 (8.93, 17.8)</td></lod>	0.182 (0.0823, 0.343)	0.0426 (0.0257, 0.0613)	0.0512 (0.0313, 0.0630)	14.6 (8.93, 17.8)		
2	4.52 (3.09, 5.79)	<lod (<lod,="" <lod)<="" td=""><td>0.0366 (0.0306, 0.0410)</td><td>0.0154 (0.0109, 0.0203)</td><td>0.0278 (0.0197, 0.0354)</td><td>4.60 (3.15, 5.88)</td></lod>	0.0366 (0.0306, 0.0410)	0.0154 (0.0109, 0.0203)	0.0278 (0.0197, 0.0354)	4.60 (3.15, 5.88)		
7	0.157 (0.0850, 0.255)	<lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" 0.0123)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" 0.0156)<="" td=""><td>0.176 (0.0980, 0.286)</td></loq></td></lod></td></loq></td></lod>	<loq (<loq,="" 0.0123)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" 0.0156)<="" td=""><td>0.176 (0.0980, 0.286)</td></loq></td></lod></td></loq>	<lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" 0.0156)<="" td=""><td>0.176 (0.0980, 0.286)</td></loq></td></lod>	<loq (<loq,="" 0.0156)<="" td=""><td>0.176 (0.0980, 0.286)</td></loq>	0.176 (0.0980, 0.286)		
14	0.112 (0.0472, 0.205)	<lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" 0.0122)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.127 (0.0602, 0.225)</td></loq></td></lod></td></loq></td></lod>	<loq (<loq,="" 0.0122)<="" td=""><td><lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.127 (0.0602, 0.225)</td></loq></td></lod></td></loq>	<lod (<lod,="" <lod)<="" td=""><td><loq (<loq,="" <loq)<="" td=""><td>0.127 (0.0602, 0.225)</td></loq></td></lod>	<loq (<loq,="" <loq)<="" td=""><td>0.127 (0.0602, 0.225)</td></loq>	0.127 (0.0602, 0.225)		
			Whole Pla	nt				
0	6.89 (5.93, 8.66)	0.0115 ( <loq, 0.0161)</loq, 	1.82 (1.16, 2.24)	0.0460 (0.0440, 0.0487)	0.0455 (0.0426, 0.0493)	8.81 (8.10, 9.92)		
1	6.19 (5.22, 6.88)	0.0137 (0.0120, 0.0155)	2.27 (2.21, 2.32)	0.0392 (0.0345, 0.0432)	0.0547 (0.0442, 0.0608)	8.57 (7.58, 9.20)		
2	6.49 (6.15, 7.00)	0.0133 (0.0130, 0.0136)	2.30 (2.19, 2.53)	0.0399 (0.0372, 0.0438)	0.0712 (0.0631, 0.0836)	8.92 (8.45, 9.33)		
7	3.77 (2.42, 4.79)	0.0109 ( <loq, 0.0158)</loq, 	1.50 (1.07, 1.94)	0.0267 (0.0191, 0.0331)	0.0876 (0.0661, 0.102)	5.39 (3.58, 6.88)		
14	1.15 (0.828, 1.39)	<loq (<loq,<br=""><loq)< td=""><td>0.450 (0.362, 0.495)</td><td>0.0148 (0.0135, 0.0157)</td><td>0.0817 (0.0699, 0.0992)</td><td>1.71 (1.28, 2.00)</td></loq)<></loq>	0.450 (0.362, 0.495)	0.0148 (0.0135, 0.0157)	0.0817 (0.0699, 0.0992)	1.71 (1.28, 2.00)		

LOQ/LOD = 0.00100/0.000300 mg/kg for sulfoxaflor in nectar; LOQ/LOD = 0.0100/0.00300 mg/kg for all remaining analytes in all remaining matrices.

Trends in sulfoxaflor and total sulfoxaflor residue concentrations declined in alfalfa pollen, nectar, and whole plant samples from the early- and early-mid bloom period (0-2 DALA) to the late-bloom period (14 DALA) at both trial locations (Figures 1-3).

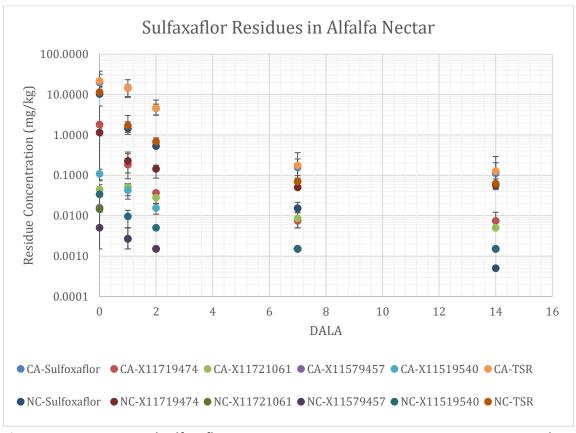


Figure 1. Mean measured sulfoxaflor, X11579457, X11719474, X11721061, X11519540 and total sulfoxaflor residues (TSR) in alfalfa nectar across study sites. Error bars represent maximum and minimum replicate values.

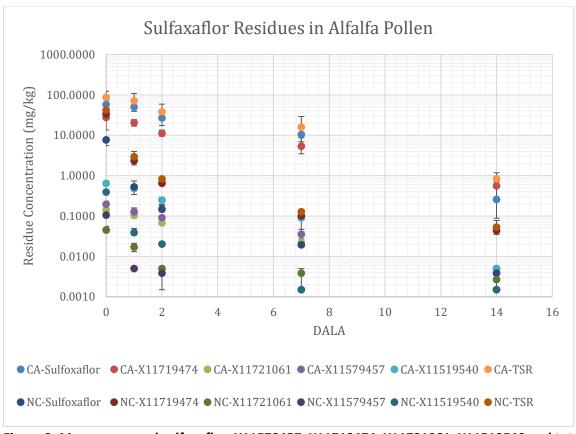


Figure 2. Mean measured sulfoxaflor, X11579457, X11719474, X11721061, X11519540 and total sulfoxaflor residues (TSR) in alfalfa pollen across study sites. Error bars represent maximum and minimum replicate values.

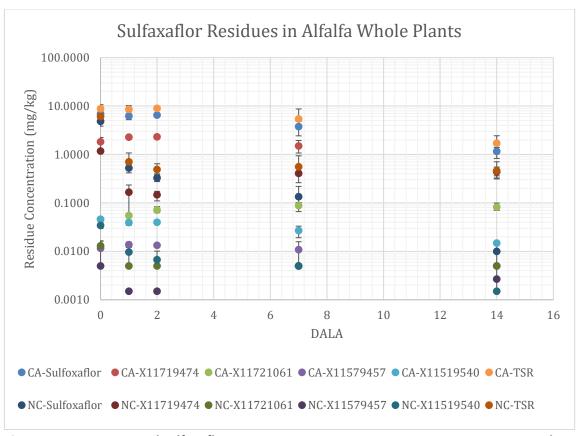


Figure 3. Mean measured sulfoxaflor, X11579457, X11719474, X11721061, X11519540 and total sulfoxaflor residues (TSR) in alfalfa whole plant across study sites. Error bars represent maximum and minimum replicate values.

#### 6C. RESIDUE DECLINE (DT<sub>50</sub>) IN ALFALFA MATRICES

For estimation of  $DT_{50}$  and  $DT_{90}$  values of sulfoxaflor in pollen and nectar, kinetic evaluation of sulfoxaflor residues data was conducted using the Computer Assisted Kinetic Evaluation (CAKE) software, version 3.3. Due to the relatively small number of sampling events over time,  $DT_{50}$  and  $DT_{90}$  values were estimated using the single first order model (SFO) to avoid overparameterization of the data sets with higher order models. Estimation of  $DT_{50}$  and  $DT_{90}$  values was done on an individual trial basis whenever possible and when replicate samples were measured within a sampling event. Prior to estimating  $DT_{50}$  and  $DT_{90}$  values, residue trial data sets were screened to ensure that sufficient data were available to produce reliable estimates (e.g., replicate values above the LOQ for 4 or more sampling events with appropriate spacing between sampling events).

The reliability of DT<sub>50</sub> estimates was evaluated based on several statistical attributes of the SFO model fit:

- statistical significance of the dissipation rate constant (k);
- correlation coefficient (r2);
- 90th percentile confidence limits around 'k'.

Due to the large degree of variability associated with pollen and nectar residue data with other pesticides, the following criteria were used to determine acceptability of DT<sub>50</sub> estimates from this analysis:

- p values for 'k' of 0.1 or less;
- r2 of 0.25 or greater; and
- 90th percentile C.L. of 'k' which did not overlap zero.

With the alfalfa residue data set, reliable DT<sub>50</sub> estimates could be estimated for both trails (NC and CA) and both the nectar and pollen matrices. Three replicate measurements were taken on each of the 5 sampling events at appropriate intervals given the relatively fast dissipation kinetics of sulfoxaflor.

Results are provided in Table 8 and Appendix 1.

Table 8. DT<sub>50</sub> and DT<sub>90</sub> values for sulfoxaflor in alfalfa matrices by study region. \*

Region	DT <sub>50</sub> Values (days)	DT <sub>90</sub> Values (days)			
Nectar from Flowers					
North Carolina	0.37	1.2			
California	1.2	4.1			
Pollen from Flowers					
North Carolina	0.26	0.87			
California	2.3	7.7			

<sup>\*</sup>DT<sub>50</sub> values were calculated following the maximum mean detection.

#### 7. STUDY STRENGTHS, LIMITATIONS AND CONCLUSIONS

In the context of documenting the magnitude of sulfoxaflor residues in bee-related matrices of alfalfa resulting from two foliar applications (the maximum seasonal treatment level), the following <u>strengths</u> are observed with this study.

- 1. Concentrations were measured for toxicologically-relevant metabolites in three bee-relevant plant matrices.
- 2. Application methods and rates were well documented.
- 3. Sampling contained an adequate amount of replication and compositing to follow EPA recommendations for studies conducted on the magnitude of residues in pollen.
- 4. Trials were conducted across two different states in two different Ecoregions (NAFTA 2 and 10). This allowed for comparison of residue magnitudes in alfalfa matrices across different soil type and climatic conditions. In addition, trials were conducted during two different growing periods (May or August).
- 5. Pre-treatment samples of whole plant did not contain detectable quantities of sulfoxaflor or any of its metabolites.

The following limitations were noted with this study:

- 1. Only two sites were evaluated, whereas USEPA (2016) recommends a minimum of 3 sites/regions within the growing area. Therefore, variability in residue values associated with geographic differences among growing regions may be underestimated.
- 2. Sugar content (Brix %) of nectar samples was not determined during the study due to insufficient sample availability.
- 3. The following overall RSDs exceeded 20% during concurrent QC analyses: sulfoxaflor in nectar

(23.2%), X11519540 in nectar (27.7%), and sulfoxaflor in whole plant (21.8%).

Overall, considering the strengths and limitations of this study, the following conclusions can be drawn:

- 1. Two foliar applications to alfalfa plants at 0.090 lb ai/A/application (based on a maximum seasonal rate of 0.186 lb ai/A), yielded detectable residues of sulfoxaflor in nectar, pollen and whole plant at both trial sites.
- 2. In alfalfa plant matrices, total sulfoxaflor residues (TSR) were greatest for each trial site in pollen, followed by nectar and then whole plant matrices, and measured residues were greater in the California trial (Trial 2) compared to North Carolina (Trial 1). In California, maximum mean TSR levels were 87.0, 21.8, and 8.92 mg/kg in pollen, nectar, and whole plant matrices, respectively, and in North Carolina, maximum mean TSR levels were 41.9, 11.5, and 6.05 mg/kg, respectively.
- 3. In nectar and whole plant samples, parent sulfoxaflor accounted for the majority of TSR while in pollen samples, sulfoxaflor accounted for the majority of TSR at the California trial, and metabolite X11719474 accounted for the majority of TSR at the North Carolina trial. Maximum mean concentrations of sulfoxaflor observed at the California trial (0 DALA) were 58.4 mg/kg in pollen, 19.8 mg/kg in nectar, and 6.89 mg/kg in whole plant. Maximum mean concentrations of sulfoxaflor at the North Carolina trial (0 DALA) were 7.67, 10.3, and 4.83 mg/kg in pollen, nectar, and whole plant samples, respectively. The major metabolite, X11719474, was present at maximum mean concentrations of 33.7 and 27.6 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively, and ranged from maximum means of 1.14 to 2.30 mg/kg in nectar and whole plant samples from both trials (0 to 2 DALA). Metabolite X11519540 was present at maximum mean concentrations of 0.394 and 0.641 mg/kg in pollen from North Carolina and California trials (each at 0 DALA), respectively. Metabolites X11579457 and X11721061 were found at maximum mean concentrations of 0.195 mg/kg and 0.142 mg/kg, respectively, in pollen from the California trial (each at 0 DALA).
- 4. Trends in sulfoxaflor and total sulfoxaflor residue (TSR) concentrations declined in alfalfa pollen, nectar, and whole plant samples from the early- and early-mid bloom period (0-2 DALA) to the late-bloom period (14 DALA) at both trial locations.
- 5. The  $DT_{50}$  values for sulfoxaflor in nectar were 0.37 (NC site) and 1.2 days (CA site). The  $DT_{50}$  values for sulfoxaflor in pollen were 0.26 (NC site) and 2.3 days (CA site).

#### 8. STUDY VALIDITY/CLASSIFICATION

Data from the two study locations are considered scientifically sound and useful for risk assessment purposes, although a minimum of 3 trials in 3 regions is recommend and some QA spike recovery samples indicated greater than expected recovery at the LOQ. Overall, this study is classified as **supplemental** for quantitative use in risk assessment.

#### 9. REFERENCES

Rodrigues Jr., A. 2011. Frozen Storage Stability of Sulfoxaflor (XDE-208) and its Main Metabolites in Crops. MRID 47832224. Unpublished study. Dow AgroSciences Study ID: 090091. June 14, 2011.

Howerton, H., and L. Gilson. 2017. Residues of Sulfoxaflor in Sunflower Nectar and Pollen after Foliar Application with GF-2372. Unpublished study. Dow AgroSciences Study ID: 150537. June 9, 2017.

USEPA 2016. Guidance on Exposure and Effects Testing for Assessing Risks to Bees. Office of Pesticide Programs, U.S. Environmental Protection Agency, July 5, 2016

## APPENDIX I. OUTPUT FROM DT50 ANALYSIS

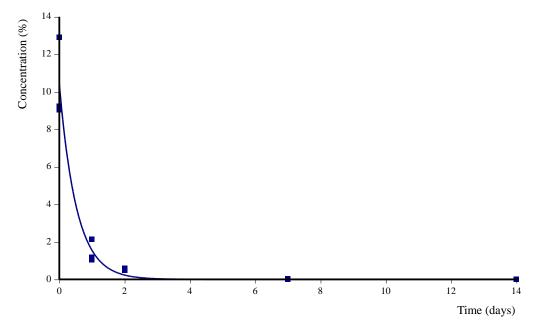
# 1. Alfalfa Nectar (2 x 0.09 lb ai/A adj. to 0.09lb ai/A) NC Trial; MRID 50444401

**CAKE Kinetic Evaluation Report** 

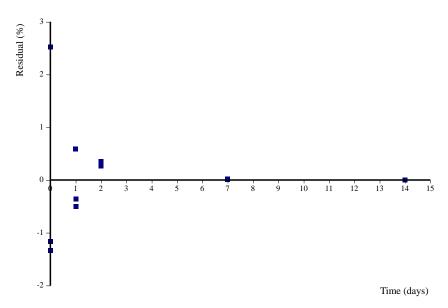
Data set: Alfalfa\_N\_0.09\_NC (SFO)

**Graphical Summary:** 

**Observations and Fitted Model:** 



#### **Residuals:**



## **Initial Values for this Step:**

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

## **Estimated Values:**

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	10.39	0.5197	N/A	9.469	11.31	9.266	11.51
k_Parent	1.903	0.3251	2.83E-005	1.327	2.479	1.201	2.605

## $\chi^2 \\$

Parameter	Error %	Degrees of Freedom
All data	4.52	3
Parent	4.52	3

## **Decay Times:**

Compartment	DT50 (days)	DT90 (days)
Parent	0.364	1.21

## **Additional Statistics:**

Parameter	r² (Obs v Pred)	Efficiency
All data	0.9579	0.9577
Parent	0.9579	0.9577

## **Parameter Correlation:**

Parent_0	k_Parent

Parent_0	1	0.1476
k_Parent	0.1476	1

### **Observed v. Predicted:**

**Compartment Parent** 

Time (days)	Value (%)	Predicted Value	Residual
0	9.05	10.39	-1.339
0	9.222	10.39	-1.167
0	12.92	10.39	2.526
1	2.139	1.549	0.5899
1	1.191	1.549	-0.3582
1	1.049	1.549	-0.5002
2	0.586	0.231	0.355
2	0.51	0.231	0.279
2	0.497	0.231	0.266
7	0.022	1.643E-05	0.02198
7	0.012	1.643E-05	0.01198
7	0.013	1.643E-05	0.01298
14	0.001	0	0.001
14	0.001	0	0.001
14	0.001	0	0.001

### **Sequence Creation Information:**

Fit generated by CAKE version 3.3 (Release) running on R version 3.0.0 (2013-04-03)

### **Report Information:**

Report generated by CAKE version 3.3 (Release) CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta Running on .NET version 4.0.30319.42000

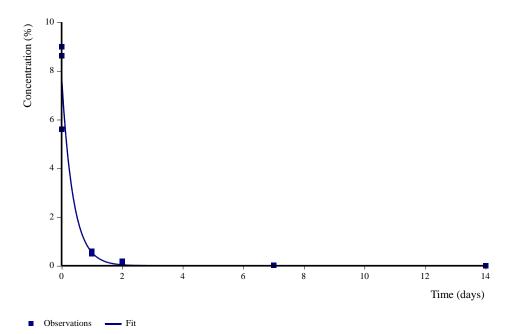
# 2. Alfalfa Pollen (2 x 0.09 lb ai/A adj. to 0.09lb ai/A) NC Trial; MRID 50444401

## **CAKE Kinetic Evaluation Report**

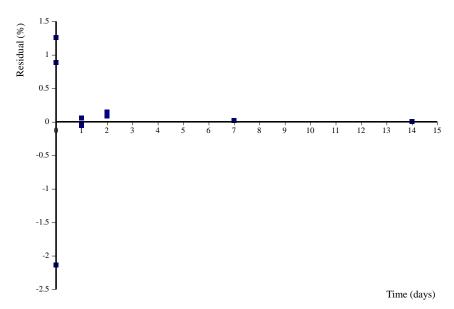
# Data set: Alfalfa\_P\_0.09\_NC (SFO)

## **Graphical Summary:**

**Observations and Fitted Model:** 



#### **Residuals:**



## **Initial Values for this Step:**

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

## **Estimated Values:**

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	7.735	0.4225	N/A	6.987	8.483	6.822	8.648
k_Parent	2.653	0.7695	0.002164	1.29	4.015	0.9904	4.315

## $\chi^2$

Parameter	Error %	Degrees of Freedom
All data	2.42	3
Parent	2.42	3

## **Decay Times:**

Compartment	DT50 (days)	DT90 (days)	
Parent	0.261	0.868	

## **Additional Statistics:**

Parameter	r² (Obs v Pred)	Efficiency
All data	0.952	0.9519
Parent	0.952	0.9519

## **Parameter Correlation:**

	Parent_0	k_Parent
Parent_0	1	0.07029
k_Parent	0.07029	1

## **Observed v. Predicted:**

**Compartment Parent** 

Time (days)	Value (%)	Predicted Value	Residual
0	5.6	7.735	-2.135
0	8.99	7.735	1.255
0	8.617	7.735	0.8819
1	0.497	0.545	-0.04801
1	0.488	0.545	-0.05701
1	0.603	0.545	0.05799
2	0.127	0.0384	0.0886
2	0.138	0.0384	0.0996
2	0.184	0.0384	0.1456
7	0.019	0	0.019
7	0.017	0	0.017

7	0.022	0	0.022
14	0.003	0	0.003
14	0.007	0	0.007
14	0.003	0	0.003

#### **Sequence Creation Information:**

Fit generated by CAKE version 3.3 (Release) running on R version 3.0.0 (2013-04-03)

#### **Report Information:**

Report generated by CAKE version 3.3 (Release) CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta Running on .NET version 4.0.30319.42000

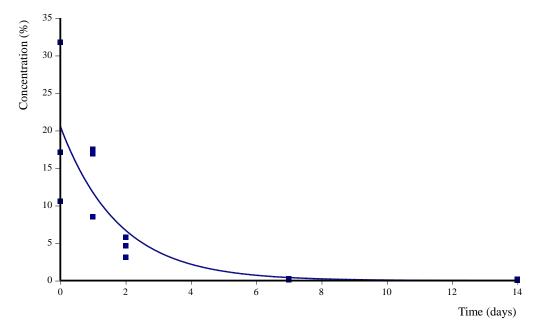
# 3. Alfalfa Nectar (2 x 0.09 lb ai/A adj. to 0.09lb ai/A) CA Trial; MRID 50444401

## **CAKE Kinetic Evaluation Report**

Data set: Alfalfa\_0.09\_N\_CA (SFO)

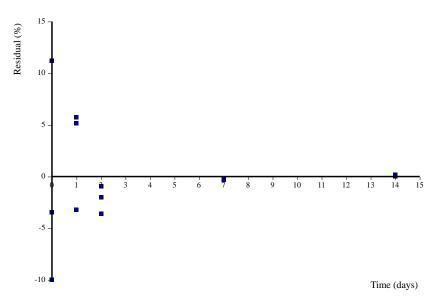
### **Graphical Summary:**

**Observations and Fitted Model:** 



Observations — Fit

### **Residuals:**



## **Initial Values for this Step:**

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

## **Estimated Values:**

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	20.55	2.815	N/A	15.57	25.54	14.47	26.63
k_Parent	0.5603	0.1867	0.005114	0.2296	0.891	0.1569	0.964

 $\chi^2$ 

Parameter	Error %	Degrees of Freedom
All data	15.9	3
Parent	15.9	3

## **Decay Times:**

Compartment	DT50 (days)	DT90 (days)	
Parent	1.24	4.11	

## **Additional Statistics:**

Parameter	r² (Obs v Pred)	Efficiency
All data	0.7367	0.7364
Parent	0.7367	0.7364

## **Parameter Correlation:**

	Parent_0	k_Parent	
Parent_0	1	0.5153	

k_Parent	0.5153	1
----------	--------	---

### **Observed v. Predicted:**

**Compartment Parent** 

Time (days)	Value (%)	Predicted Value	Residual
0	17.08	20.55	-3.473
0	10.59	20.55	-9.966
0	31.77	20.55	11.21
1	16.88	11.74	5.144
1	17.48	11.74	5.744
1	8.521	11.74	-3.216
2	4.675	6.702	-2.027
2	3.087	6.702	-3.615
2	5.784	6.702	-0.9183
7	0.13	0.407	-0.277
7	0.255	0.407	-0.152
7	0.085	0.407	-0.322
14	0.047	0.008058	0.03894
14	0.205	0.008058	0.1969
14	0.082	0.008058	0.07394

### **Sequence Creation Information:**

Fit generated by CAKE version 3.3 (Release) running on R version 3.0.0 (2013-04-03)

### **Report Information:**

Report generated by CAKE version 3.3 (Release)

CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta

Running on .NET version 4.0.30319.42000

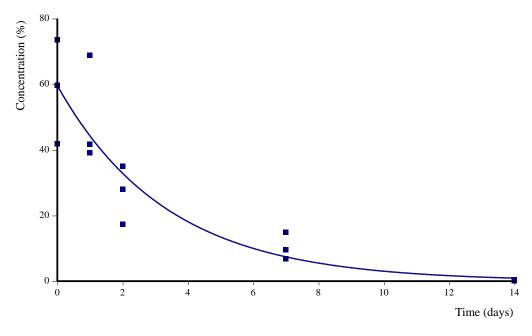
# 4. Alfalfa Pollen (2 x 0.09 lb ai/A adj. to 0.09lb ai/A) CA Trial; MRID 50444401

## **CAKE Kinetic Evaluation Report**

# Data set: Alfalfa\_P\_0.09\_CA (SFO)

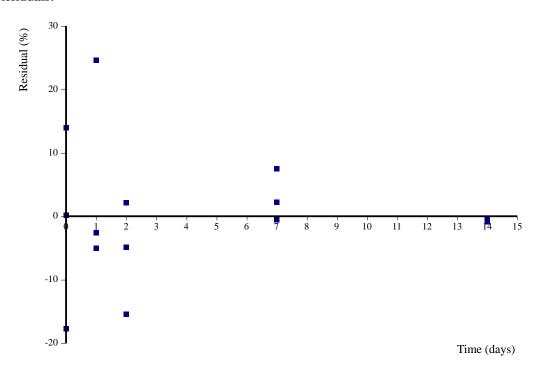
## **Graphical Summary:**

**Observations and Fitted Model:** 



■ Observations — Fit

### **Residuals:**



## **Initial Values for this Step:**

Parameter	Initial Value	Bounds	Fixed
Parent_0	100	0 to (unbounded)	No
k_Parent	0.1	0 to (unbounded)	No

## **Estimated Values:**

Parameter	Value	σ	Prob. > t	Lower (90%) CI	Upper (90%) CI	Lower (95%) CI	Upper (95%) CI
Parent_0	59.58	5.516	N/A	49.81	69.34	47.66	71.49
k_Parent	0.2977	0.07895	0.001168	0.1579	0.4375	0.1271	0.468

 $\chi^2 \\$ 

Parameter	Error %	Degrees of Freedom
All data	11	3
Parent	11	3

## **Decay Times:**

Compartment	DT50 (days)	DT90 (days)	
Parent	2.33	7.74	

## **Additional Statistics:**

Parameter	r² (Obs v Pred)	Efficiency
All data	0.8296	0.8295
Parent	0.8296	0.8295

### **Parameter Correlation:**

	Parent_0	k_Parent
Parent_0	1	0.5793
k_Parent	0.5793	1

#### **Observed v. Predicted:**

**Compartment Parent** 

Time (days)	Value (%)	Predicted Value	Residual
0	41.85	59.58	-17.72
0	59.73	59.58	0.1586
0	73.52	59.58	13.94
1	39.16	44.24	-5.081
1	68.82	44.24	24.59
1	41.65	44.24	-2.583
2	34.96	32.85	2.113
2	17.38	32.85	-15.47
2	27.97	32.85	-4.879
7	9.629	7.415	2.214
7	6.882	7.415	-0.5328
7	14.88	7.415	7.468
14	0.184	0.9229	-0.7389
14	0.503	0.9229	-0.4199
14	0.088	0.9229	-0.8349

### **Sequence Creation Information:**

Fit generated by CAKE version 3.3 (Release) running on R version 3.0.0 (2013-04-03)

#### **Report Information:**

Report generated by CAKE version 3.3 (Release) CAKE developed by Tessella Ltd, Abingdon, Oxfordshire, UK, sponsored by Syngenta Running on .NET version 4.0.30319.42000